

## PATENT ABSTRACTS OF JAPAN

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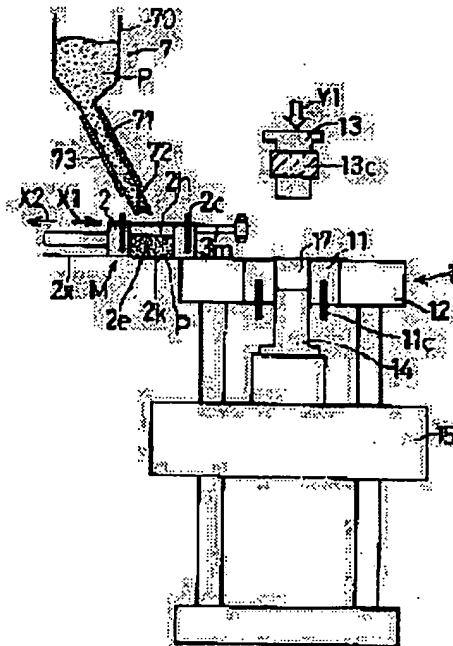
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## (54) POWDER MOLDING METHOD

## (57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a method for molding powder by which a green compact of high density can be obtained.

**SOLUTION:** A molding die 1 having a mold surface dividing a molding cavity 17 and powder for molding P not containing lubricant are used and a heating operation in which both the molding die 1 and the powder for molding P are heated at 150 to 400° C are performed and, further, an operation to apply the lubricant on the mold surface of the molding die 1 is performed. The heated powder for molding P is charged in the heated molding cavity 17. The powder for molding P is compression-molded by an upper punch mold 13 to obtain the green compact.



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技術表示箇所

審査請求 未請求 請求項の数1 OL (全 8 頁)

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## 【特許請求の範囲】

【請求項1】 成形キャビティを区画する型面を備えた粉末成形用の成形型と、潤滑剤を実質的に含有しない成形用粉末とを用い、

前記成形型及び前記成形用粉末を150～400℃の温度領域に加熱する加熱操作を行うと共に、前記成形型の型面に前記潤滑剤を塗布する塗布操作とを行う工程と、

前記温度領域に加熱された前記成形型の成形キャビティに、前記温度領域に加熱された前記成形用粉末を装填する工程と、

前記成形キャビティに装填した前記成形用粉末を圧縮成形して圧粉体とする工程とを順に行うことを特徴とする粉末成形方法。

## 【発明の詳細な説明】

【0001】

【発明の属する技術分野】 本発明は粉末を圧縮成形して圧粉体を得る粉末成形方法に関する。

【0002】

【従来の技術】 従来より粉末冶金では、ステアリン酸亜鉛等の潤滑剤が成形用粉末に添加されている。これにより成形用粉末の潤滑性が確保されている。また米国特許公報4,955,798号には、固体潤滑剤を含有する粉末冶金用の成形用粉末を用い、その成形用粉末を、常温以上でかつ固体潤滑剤が溶ける温度以下の温度領域、つまり70℃以上で150℃未満の温度領域、一般的には70～120℃の温度領域に加熱し、その加熱した成形用粉末を用いて、成形型で圧縮成形して圧粉体を形成する温間粉末成形方法が開示されている。

【0003】 また特開平5-271709号公報には、10～30wt%の $C_{10}\sim C_{22}$ であるモノカルボン酸と、10～30wt%の $C_{10}\sim C_{22}$ であるジカルボン酸と、40～80wt%の $(CH_2)_x$ 、 $(CH_2)_x$  (x:2～6)であるジアミンとの反応生成物である高温用潤滑剤を成形用粉末に添加する技術が開示されている。この潤滑剤によれば、370℃以下で粉末冶金用潤滑剤として使用可能とされている。

【0004】

【発明が解決しようとする課題】 ところで粉末粒子自体の高密度化を図るには、粉末の加熱温度を高くすればよい。しかし成形用粉末を150℃以上に加熱すれば、成形用粉末に添加されている潤滑剤の流動性が著しく低下し、高い密度をもつ圧粉体を得ることができない。上記した米国特許公報4,955,798号に係る技術においても、150℃未満であれば、成形用粉末の流動性が確保されるものの、150℃以上に加熱すれば、潤滑剤が融け、成形用粉末の流動性が著しく低下するため、粉箱等を使った粉末充填ができず、連続成形（生産性）の面で不利である。

【0005】 また特開平5-271709号公報に係る

技術によれば、一般的な潤滑剤に比較して融点が高いため370℃以下で使用可能ではあるものの、やはり150℃以上では、粉末の流動性が低下する。上記した事情により、従来に係る温間粉末成形方法によれば、粉末の加熱温度は150℃未満となっていた。

【0006】 本発明は上記した実情に鑑みなされたものであり、その課題は、圧粉体の密度や強度を向上するのに有利な粉末成形方法を提供することにある。

【0007】

【課題を解決するための手段】 本発明者は上記課題のもとに鋭意開発を進め、潤滑剤を実質的に含有しない成形用粉末を用い、成形用粉末及び成形型の双方を150～400℃の温度領域に加熱すると共に、成形型の型面に潤滑剤を塗布した状態で、成形用粉末を圧縮成形して圧粉体を成形すれば、圧粉体の密度や強度を向上するのに有利であることを知見し、本発明方法を開発した。

【0008】 即ち、本発明に係る粉末成形方法は、成形キャビティを区画する型面を備えた粉末成形用の成形型と、潤滑剤を実質的に含有しない成形用粉末とを用い、成形型及び成形用粉末とを150～400℃の温度領域に加熱する加熱操作を行うと共に、成形型の型面に潤滑剤を塗布する塗布操作とを行う工程と、前記温度領域に加熱された成形型の成形キャビティに、前記温度領域に加熱された成形用粉末を装填する工程と、成形キャビティに装填した成形用粉末を圧縮成形して圧粉体とする工程とを順に行うことを特徴とするものである。

【0009】

【発明の実施の形態】 本発明方法で用いる成形型は、成形キャビティを区画する型面を備えたものであり、一般的には金型を採用でき、場合によってはセラミックス型等を採用できる。本発明方法で用いる成形用粉末としては、潤滑剤を実質的に含有しないものであり、一般的には金属粉末を採用できる。従って本発明方法で用いる成形用粉末には、一般的には、潤滑剤が全く含有されていない。金属粉末の材質としては、従来より公知のもの、例えば鉄系、Ni系、銅系、アルミ系等を採用できる。金属粉末としては噴霧粉、電解粉、還元粉等の公知のものを採用できる。

【0010】 成形用粉末が高温で加熱されると、成形用粉末の軟化性、成形性が向上するものの、雰囲気が大気中であれば、成形用粉末の酸化が促進されるおそれがある。また成形用粉末の加熱温度が低温であれば、成形用粉末の酸化の抑制には有利であるものの、成形用粉末の軟化性、成形性が低下する。また成形用粉末の粉末粒子のサイズが小さいと、成形用粉末の比表面積が大きくなり、成形用粉末が活性化し、酸化し易くなる。

【0011】 この様な事情を考慮して、成形用粉末の加熱温度を決定する。成形用粉末の材質、粉末粒子の粒径等にも依るが、成形用粉末の加熱温度は、上限値としては350℃、300℃、250℃、200℃に

でき、下限値としては170°C、200°C、230°Cにできるが、これに限定されるものではないことは勿論である。

【0012】また成型型の加熱温度が高温となると、成型用粉末の成形性が向上するものの、成型型の寿命の点で不利となる。また成型型の加熱温度が低温であれば、成型型の寿命の点で有利であるものの、成型用粉末の成形性が充分でなくなるおそれがある。このような事情を考慮して、成型型の加熱温度を決定する。成型型の材質等にも依るが、成型型の加熱温度は、上限値としては350°C、300°C、250°C、200°Cにでき、下限値としては170°C、200°C、230°Cにできる。

【0013】本発明方法によれば、成型用粉末が鉄系、成型型が鉄系であれば、上記事情を考慮すると、成型用粉末及び成型型の加熱温度は150~300°C程度が好ましいが、これに限定されるものではないことは勿論である。成型用粉末を加熱する形態、成型型を加熱する形態としては、赤外線加熱、ヒータ加熱、誘導加熱、ジュール熱加熱等を採用できる。

【0014】本発明方法によれば、成型型及び成型用粉末を150~400°Cの温度領域に加熱する。成型型の加熱温度と、成型用粉末の加熱温度とは実質的に等温領域でも良いし、或いは異なっても良い。成型用粉末の加熱温度が成型型の加熱温度よりも高い場合には、成型型の寿命を確保しつつ成型用粉末の成形性を確保するのに有利であり、ひいては圧粉体の密度確保に有利である。

【0015】また成型型の加熱温度が成型用粉末の加熱温度よりも高い場合には、成型用粉末の熱が成型型に伝達されることを抑制できるため、成型用粉末の温度を維持するのに有利であり、この意味において圧粉体の密度確保に有利である。特に圧粉体の表層部分の熱が確保されるので、圧粉体の表面温度の維持に有利であり、圧粉体の表面の緻密化に有利である。

【0016】成型用粉末の加熱雰囲気としては、成型用粉末の種類に応じて適宜選択できる。大気中でも良いし、非酸化性雰囲気でも良い。非酸化性雰囲気としては減圧雰囲気、真空雰囲気、窒素雰囲気、希ガス雰囲気等を採用でき、場合によっては還元性雰囲気でも良い。成型型の加熱雰囲気についても同様である。本発明方法によれば、成型型の型面に潤滑剤が塗布される。これにより成型用粉末と成型型の型面との摩擦は低減され、成型用粉末の装填性が向上する。潤滑剤としては公知のものを採用でき、固体潤滑剤でも良いし、固体潤滑剤を溶媒に分散または溶解させたものを採用でき、例えばステアリン酸亜鉛系、黒鉛系、窒化ボロン系、金属せっけん系などを採用できる。溶媒としては水、アルコール等を採用できる。塗布手段としてはスプレー塗布、はけ塗り、浸漬等の公知の方法を採用できる。

【0017】本発明方法によれば、成型型及び成型用粉末を150~400°Cの温度領域に加熱する加熱操作と、成型型の型面に潤滑剤を塗布する塗布操作とが行われる。この場合には、加熱操作の後に塗布操作が行われても良いし、或いは、塗布操作の後に加熱操作が行われても良いし、或いは、加熱操作と塗布操作とが時間的に同時に行われても良い。

【0018】

【実施例】以下、本発明に係る実施例について説明する。まず説明の便宜上、図1を参照して成型型1から説明する。成型型1は、ダイス孔をもち固定型として機能するリング状をなす金属製のダイス型11と、ダイス型11の外周部を保持するリング状をなす金属製のダイスホルダ12と、ダイス型11に対して昇降可能な金属製の上下パンチ型13と、ダイス型11のダイス孔に嵌合された金属製の下パンチ型14とを備えている。下パンチ型14は基部15に保持されている。

【0019】ダイス型11のダイス孔の型面、下パンチ型14の型面、上パンチ型13の型面により、成形キャビティ17が区画される。上パンチ型13には、これを加熱する第1ヒータ13cが装備されている。ダイス型11には、これを加熱する第2ヒータ11cが装備されている。更にダイスホルダ12及びダイス型11の上方をガイド2xに沿って移動可能な可動式の粉箱2が装備されている。粉箱2は、成型用粉末を収容する収容室2eをもつ。収容室2eは、上面開口2h及び下面開口2kをもつ。粉箱2には、潤滑剤を噴霧するためのスプレーノズル3がアーム3mを介して装備されている。粉箱2には、収容室2e内の成型用粉末Pを加熱する第3ヒータ2cが装備されている。

【0020】また成型型1の近傍には粉末供給装置7が設けられている。粉末供給装置7は、成型用粉末Pを収容する粉末容器70と、粉末容器70から下方に延設され待機中の粉箱2の収容室2eに臨む送給路71と、送給路71を開閉する弁72と、送給路71内の成型用粉末Pを加熱する第4ヒータ73とをもつ。第1ヒータ13c、第2ヒータ11c、第3ヒータ2c、第4ヒータ73は図略の温度制御装置により制御され、それぞれの目標加熱温度に維持される。

【0021】さて本実施例によれば使用の際には、第2ヒータ11cによりダイス型11が所定の温度領域に加熱されている。第1ヒータ13cにより上パンチ型13が所定の温度領域に加熱されている。粉末供給装置7の第4ヒータ73により粉末容器70の成型用粉末Pは所定の温度領域に加熱されている。本実施例によれば、成型型1の目標加熱温度は150~400°Cのうちの所定の温度領域である。成型用粉末Pの目標加熱温度は150~400°Cのうちの所定の温度領域である。

【0022】図1に示すように、成形キャビティ17から離れた待機位置Mに、粉箱2が待機している。粉末供

給装置7の弁72が開放すると、加熱された成形用粉末Pが粉末供給装置7から自然落下して粉箱2の収容室2eに送給される。粉箱2の収容室2e内の成形用粉末は、第3ヒータ2cにより加熱され、所定の温度領域に維持された状態で収容室2e内で待機している。

【0023】所定の時刻になると、図2(A)に示すように粉箱2がシリンダ2pにより矢印X1方向に移動する。よって図2(B)に示すようにスプレーノズル3が成形キャビティ17及び上パンチ型13に対面し、その位置で粉箱2が停止する。この状態で、スプレーノズル3が作動して、スプレーノズル3から潤滑剤Lが上下方向に噴霧される。これによりダイス型11の型面、下パンチ型14の型面、上パンチ型13の型面にそれぞれ、潤滑剤Lが塗布される。つまり成形型1の型面に潤滑剤Lを塗布する『型潤滑』操作が実行される。

【0024】本実施例によれば、ダイス型11や上パンチ型13は高温状態に加熱されているため、これらの熱で液状の潤滑剤Lは速やかに乾燥する。よって、潤滑膜がダイス型11の型面、下パンチ型14の型面、上パンチ型13の型面にそれぞれ積層される。従って、液状の潤滑剤Lを乾燥させる乾燥操作を廃止または簡略化するの

に有利であり、生産能率の向上に貢献できる。

【0025】『型潤滑』操作による塗布が終了すると、粉箱2が矢印X1方向に更に移動し、図2(C)に示すように、粉箱2の収容室2eの下面開口2kが成形キャビティ17に対面する。すると粉箱2の収容室2eの成形用粉末Pは、重力により自然落下して成形キャビティ17に装填される。装填が完了したら、粉箱2は矢印X2方向に移動して待機位置Mに戻る。その後、上パンチ型13が矢印Y1方向に下降して成形キャビティ17に押入される。よって、成形キャビティ17内の成形用粉末Pが圧縮成形され、圧粉体が成形される。

【0026】本実施例によれば、成形キャビティ17内の成形用粉末Pと成形型1の型面との摩擦は、上記した潤滑膜により低減され、これにより成形用粉末Pの装填性、圧縮成形性が向上する。上記のように圧粉体が成形されたら、次に、その圧粉体を焼結温度域に加熱して焼結体とする焼結工程、焼結体を鍛造型で熱間鍛造して焼結鍛造体とする鍛造工程が実施される。

【0027】本実施例によれば、成形用粉末Pや成形型1を加熱する際には、成形用粉末Pの加熱温度と成形型1の加熱温度とが実質的に同一の形態でも良い。また成形用粉末Pの加熱温度が成形型1の加熱温度よりも高い形態でも良い。また成形型1の加熱温度が成形用粉末Pの加熱温度よりも高い形態でも良い。成形用粉末Pの加熱温度が成形型1の加熱温度よりも高い場合には、成形用粉末Pの成形性を確保するのに有利である。ひいては圧粉体の密度確保に有利である。

【0028】成形型1の加熱温度が成形用粉末Pの加熱温度よりも高い場合には、成形用粉末Pが成形型1の型

面に接触しても、成形用粉末Pの熱が成形型1に逃げるのが抑制されるため、成形用粉末Pの温度を維持するのに有利であり、この意味において圧粉体の密度確保に有利である。特に成形型1の型面に接触する圧粉体の表面部分の温度が確保されるので、圧粉体の表面温度の維持に有利であり、表面ポアの低減、回避に有利である。

【0029】なお上記した例では、潤滑剤Lを噴霧塗布する塗布操作、粉箱2の成形用粉末Pを自然落下により成形キャビティ17に装填する装填操作は、粉箱2を停止させて行っているが、これに限らず、粉箱2を停止せずに低速で連続的に移動させることにより上記塗布操作や装填操作を行うことにしても良い。また上記した実施例では、圧粉体を焼結した焼結体を鍛造する鍛造工程が実施されるが、これに限らず、圧粉体の用途等によっては鍛造工程は省略することもできる。

【0030】また図3に示す例のようにしても良い。即ち、粉箱2の収容室2eの上面開口2hを開閉する開閉蓋2r、及び、下面開口2kを開閉する開閉蓋2sを設けると共に、収容室2eを減圧装置9に減圧路9n及び開閉弁9vを介して接続することにしても良い。減圧路9nとして、ゴムホースや蛇腹管等のように伸長可能または屈曲可能な可撓管を採用すれば、粉箱2の移動に対応できる。減圧装置9としては例えば真空ポンプを採用できる。

【0031】そして収容室2e内の成形用粉末を第3ヒータ2cで加熱しつつ、減圧装置9で収容室2eを減圧状態または真空状態とし、この状態で待機する。待機時間が終了したら、粉箱2が矢印X1方向に移動すると共に、粉箱2が所定の位置に到達したら、開閉蓋2r及び開閉蓋2sが開放して、場合によっては開閉蓋2sのみが開放するだけで、粉箱2内の成形用粉末Pは成形キャビティ17に落下する。これによれば、待機中において収容室2e内は減圧状態または真空状態とされているため、高温の成形用粉末Pの酸化を抑制するのに有利である。

【0032】このように成形用粉末Pの酸化抑制に有利であるため、成形用粉末Pの加熱温度を一層高くするのに有利であり、ひいては圧粉体の一層の高密度化に貢献でき、圧粉体や焼結体や焼結鍛造体の品質確保に貢献できる。更に図3に示すように、粉末供給装置7の粉末容器70にこれを開閉する開閉蓋70sを設けると共に、粉末容器70を減圧装置9に減圧路9m及び開閉弁9wを介して接続しても良い。この場合には、粉末容器70内の高温の成形用粉末の酸化を抑えるのに有利である。

【0033】(試験例) 本発明方法の効果を確認するために次の試験を行った。

(試験例1)

①この試験によれば、鉄系粉末と黒鉛粉末とを混合した成形用粉末を用い、圧粉体を成形する成形面圧を実施例及び比較例共に7 ton/cm<sup>2</sup>と同一値に設定している。

そして、実施例及び比較例に係る圧粉体を形成し、各圧粉体の密度を測定した。密度の測定は、日本粉末冶金工業会規格 (JPMA M 01-1992) に基づいた。なお成形用粉末における黒鉛粉末の割合は、実施例及び比較例共に、重量比で5%である。

【0034】鉄系粉末としては実施例及び比較例ともに同種のもの、つまり商品名: Distaloy AE (ヘガネス社) を採用している。Distaloy AE の目標組成は、重量比で、Fe-4%Ni-1.5%Cu-0.5%Moである。Distaloyの粒度分布は、150 $\mu$ m以上が8%、45 $\mu$ m以下が20%、45~150 $\mu$ mが残部である。

【0035】実施例1によれば、潤滑剤が添加されていない上記した成形用粉末を用い、型潤滑すると共に、成形温度を150 $^{\circ}$ Cとした状態で、つまり成形用粉末及び成形型の双方の目標温度を150 $^{\circ}$ Cとした状態で、圧粉体を成形した。実施例2によれば、潤滑剤が添加されていない上記した成形用粉末を用い、前述同様に型潤\*

\* 滑すると共に、成形温度を180 $^{\circ}$ Cとした状態で、つまり成形用粉末及び成形型の双方の温度を180 $^{\circ}$ Cとした状態で、圧粉体を成形した。

【0036】比較例1によれば、潤滑剤としてステアリン酸亜鉛を上記鉄系粉末に添加した成形用粉末を用い、成形温度を室温に維持した状態、つまり成形用粉末及び成形型の双方の温度を室温に維持した状態で圧粉体を成形した。比較例1によれば、成形用粉末を100%としたときステアリン酸亜鉛は0.5%添加した。比較例2によれば、温間成形用潤滑剤 (米国特許4955798に係る潤滑剤) を上記鉄系粉末に添加した成形用粉末を用い、成形温度を150 $^{\circ}$ Cとした状態で、つまり成形用粉末及び成形型の双方の温度を150 $^{\circ}$ Cにした状態で圧粉体を成形した。比較例1によれば、成形用粉末を100%としたとき温間成形用潤滑剤は0.5%添加した。

【0037】

【表1】

	潤滑	成形温度
比較例1	ステアリン酸亜鉛	室温
比較例2	温間成形用潤滑剤	150 $^{\circ}$ C
実施例1	型潤滑	150 $^{\circ}$ C
実施例2	型潤滑	180 $^{\circ}$ C

圧粉体の密度測定の試験結果は図4に示されている。図4から理解できるように、成形面圧が同一であっても、比較例1、2では圧粉体の密度が低いものの、実施例1、2では高密度の圧粉体が得られる。特に成形温度が180 $^{\circ}$ Cである実施例2によれば、圧粉体密度が最も高い。従って本実施例方法によれば、圧粉体を得る際の成形面圧が同一 (=7 ton/cm $^2$ ) であっても、圧粉体の密度を増加できることがわかる。その理由は、潤滑剤がないため気孔の緻密化が良好であるためと推察される。

【0038】②更に圧粉体の密度を7.1 [g/cm $^3$ ] に設定したときにおける実施例及び比較例に係る圧粉体の抗折強度を測定した。抗折強度の測定は、日本粉末冶金工業会規格 (JPMA M 09-1992) に基づいた。試験結果を図5に示す。図5から理解できるように、比較例1、2では圧粉体の強度が低いものの、実施例1では高強度つまり3 [kg/cm $^2$ ] を越える強度の圧粉体が得られた。従って本実施例方法によれば、圧粉体密度が同一であっても、圧粉体の強度を増加できることがわかる。その理由は、潤滑剤がないため粉末粒子間の固着 (粉のかみあい) がより強固になるものと推察されている。

【0039】(試験例2)

①この試験によれば、実施例及び比較例共に、鉄系粉末と黒鉛粉末とを混合した成形用粉末を圧縮成形して圧粉体を成形する際の成形面圧を5 ton/cm $^2$ 、6 ton/cm $^2$ 、7 ton/cm $^2$ と変更して成形したとき、圧粉体の密度変化を調べた。密度の測定は前述同様に、日本粉末冶金工業会規格 (JPMA M 01-1992) に基づいた。なお成形用粉末における黒鉛粉末の割合は、前述同様に実施例及び比較例共に、重量比で5%である。

【0040】実施例Aによれば、鉄系粉末として前述のDistaloy AE (ヘガネス社) を採用し、粉末及び成形型の双方の目標温度を共に150 $^{\circ}$ Cとした。Distaloy AE の目標組成は前述したように、重量比で、Fe-4%Ni-1.5%Cu-0.5%Moである。実施例Aによれば、測定結果を図6に示す。図6の横軸は成形面圧を示し、縦軸は圧粉体の密度を示す。図6においてRTは室温を意味する。

【0041】比較例Bによれば、鉄系粉末としてDistaloy AE (ヘガネス社) を用い、粉末及び成形型の温度を共に室温としている。比較例Cでは、鉄系粉末としてステアリン酸亜鉛を添加したDistaloy AE (ヘガネス社)

を用い、成形用粉末及び成形型の温度を共に室温としている。比較例Dによれば、ステアリン酸亜鉛に代えて高温領域でも溶融しない高温用潤滑剤を添加したAncordense（ヘガネス社）を鉄系粉末として用い、粉末及び成形型の温度を共に室温としている。

【0042】比較例Eによれば、Ancordenseを鉄系粉末として用い、粉末及び成形型の温度を共に室温としている。図6から理解できるように、粉末及び成形型の双方が室温領域で圧粉体を成形した比較例C、比較例D、比較例Bによれば、圧粉体の密度が低い。また成形用粉末に高温用潤滑剤が添加されている比較例Eによれば、成形面圧が5 [ton/cm<sup>2</sup>] ~ 6 [ton/cm<sup>2</sup>] では圧粉体の密度が高いものの、成形面圧が7 [ton/cm<sup>2</sup>] に近づくと、密度向上効果は飽和状態に近くなっている。

【0043】これに対して粉末及び成形型の双方の目標温度が150°Cである実施例Aによれば、成形面圧が7 [ton/cm<sup>2</sup>] となっても、密度向上効果は飽和状態ではない。すなわち図6から理解できる様に実施例Aによれば、成形面圧が7 [ton/cm<sup>2</sup>] あれば、密度は比較例Eに係る密度よりも高い。その理由は、成形面圧を高くしたとしても、潤滑剤を含む場合には、潤滑剤に相当するぶんは金属粉末が実質的に装填されず、実質的に空孔とされ、高密度の圧粉体が得られないものの、潤滑剤が無ければ、そのような不具合がないためであると推察される。

②また上記した実施例A～比較例Eに係る圧粉体について、抗折強度を調べる試験を行った。抗折強度の測定は、日本粉末冶金工業会規格（JPMA M 09-1992）に基づいた。試験結果を図7に示す。図7は横軸が圧粉体の密度を示し、縦軸が圧粉体の強度を示す。実施例Aによれば、圧粉体の密度が7.1 [g/cm<sup>3</sup>] ~ 7.3 [g/cm<sup>3</sup>] ~と高くなれば、圧粉体の強度も3 [kgf/mm<sup>2</sup>] 付近～4.7 [kgf/mm<sup>2</sup>] 付近へと向上する。即ち実施例Aによれば、特性線の上昇勾配は大きい。

【0044】しかし比較例Eによれば、圧粉体の密度が7.1 [g/cm<sup>3</sup>] ~ 7.3 [g/cm<sup>3</sup>] ~と高くなったとしても、圧粉体の強度は2.5 [kgf/mm<sup>2</sup>] 付近からあまり向上していない。即ち比較例Eによれば、特性線の上昇勾配は小さい。

③ところで使用の際には、上記した圧粉体は、焼結されて焼結体とされる。更に密度を高めるために、焼結後に鍛造で強圧して焼結鍛造体とすることが好ましい。この場合実際の操業では、焼結体を焼結工程から鍛造工程に移送する必要があるため、移送中に焼結体は大気に触れる。大気に触れる度合いが高い状態の焼結体が鍛造されると、焼結鍛造体に表面欠陥が発生し、焼結鍛造体の強度確保のために不利であることは既に知られている。

【0045】そこでこの試験では、上記した圧粉体を焼結温度域（1120～1150°C）で焼結して焼結体

を形成し、その焼結体を大気に所定時間T（T=10秒間）露出し、その後、その焼結体を熱間鍛造して焼結鍛造体とした。そして焼結鍛造体の表面欠陥の面積率を測定し、圧粉体の密度 $\rho$ をパラメータとして、図8に示した。

【0046】図8は横軸が焼結鍛造体の表面からの距離を示し、縦軸が表面欠陥の面積率を意味する。図8から理解できるように、表面欠陥の面積率は圧粉体の密度に大いに影響を受ける。即ち、圧粉体が低密度（ $\rho=5.8$ ）であれば、焼結鍛造体の表面欠陥の面積率は高い。表面の緻密化が低下しており、焼結体の表面酸化が進行し易いためと推察される。しかし圧粉体が高密度（ $\rho=6.8$ ）であれば、焼結鍛造体の表面欠陥の面積率は低い。表面の緻密化が進んでおり、焼結体の表面酸化が進行しにくいと推察される。

【0047】従って圧粉体の密度を高めに行ける効果が得られる本発明方法によれば、焼結鍛造体の表面欠陥を低減するのに有利である。故に焼結鍛造体の強度増加に有利である。また図9は、圧粉体の密度と焼結鍛造体の表面欠陥の面積率との関係を示す。図9の特性線から理解できるように、圧粉体の密度が低くなれば、焼結鍛造体の表面欠陥の面積率が増加し、圧粉体の密度が高くなれば、焼結鍛造体の表面欠陥の面積率が低下することがわかる。

【0048】（付記）上記した実施例から次の技術的思想も把握できる。

①請求項1において、成形用粉末と成形型とは実質的に等温領域であることを特徴とする粉末成形方法。

②請求項1において、成形用粉末の温度<成形型の温度であることを特徴とする粉末成形方法。

③請求項1において、成形用粉末の温度>成形型の温度であることを特徴とする粉末成形方法。

④成形型から離れた待機位置と成形型に対面する位置との間で移動可能な粉箱を用い、待機中の粉箱内は減圧状態または真空状態に維持可能とされていることを特徴とする粉末成形方法。

⑤成形キャビティを区画する型面を備えた粉末成形用の成形型と、潤滑剤を実質的に含有しない成形用粉末とを用い、成形型及び成形用粉末とを150～400°Cの温度領域に加熱すると共に、成形型の型面に潤滑剤を塗布する工程と、成形型の成形キャビティに成形用粉末を装填する工程と、成形キャビティに装填した成形用粉末を圧縮成形して圧粉体とする工程と、圧粉体を焼結して焼結体とする工程と、焼結体を鍛造して焼結鍛造体とする工程とを順に行うことを特徴とする焼結鍛造体の製造方法。圧粉体の高密度化を図り得るため、焼結鍛造体の表面欠陥を低減するのに有利であり、焼結鍛造体の強度確保に貢献できる。

【0049】

【発明の効果】本発明方法によれば、成形キャビティを

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区画する型面を備えた粉末成形用の成型型と、潤滑剤を実質的に含有しない成形用粉末とを用い、成型型及び成形用粉末とを $150\sim 400^{\circ}\text{C}$ の温度領域に加熱すると共に、成型型の型面に潤滑剤を塗布する工程と、成型型のキャビティに成形用粉末を装填する工程とを順に行う。そのため圧粉体の密度、強度を高めるのに有利である。

【0050】このように圧粉体の密度を高くすれば、圧粉体の表面のポアの低減に有利である。従って圧粉体を焼結鍛造した場合に、焼結鍛造体の表面欠陥の低減に有利であり、焼結鍛造体の強度の向上に一層有利である。

【図面の簡単な説明】

【図1】実施例に係る構成図である。

【図2】実施例に係る各工程を示す構成図である。

【図3】他の実施例に係る構成図である。

【図4】成形面圧を7〔ton/cm<sup>2</sup>〕とした場合における\*

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\* 圧粉体密度を示すグラフである。

【図5】成型型密度を7.1〔g/cm<sup>3</sup>〕とした場合における圧粉体強度を示すグラフである。

【図6】成形面圧と圧粉体密度との関係を示すグラフである。

【図7】圧粉体密度と圧粉体強度との関係を示すグラフである。

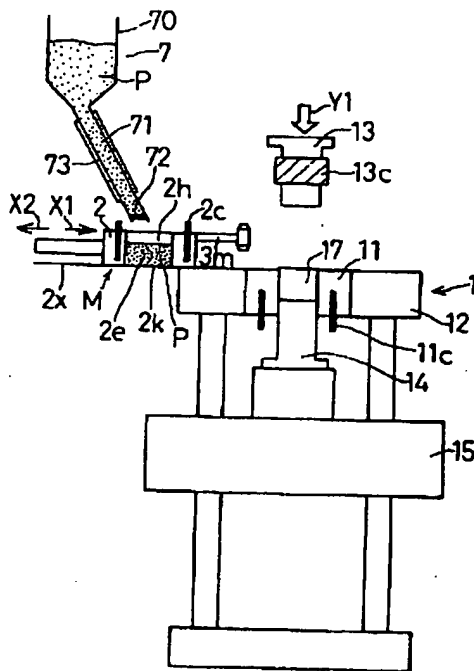
【図8】焼結鍛造体の表面からの距離と表面欠陥面積率との関係を示すグラフである。

【図9】圧粉体密度と焼結鍛造体の表面欠陥面積率との関係を示すグラフである。

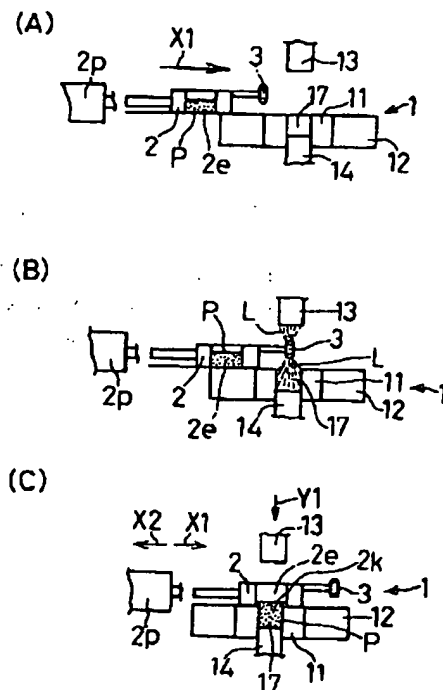
【符号の説明】

図中、1は成型型、11はダイス型、13は上パンチ型、17は成形キャビティ、11c、12c、13c、73はヒータ、2は粉箱、3はスプレーノズルを示す。

【図1】

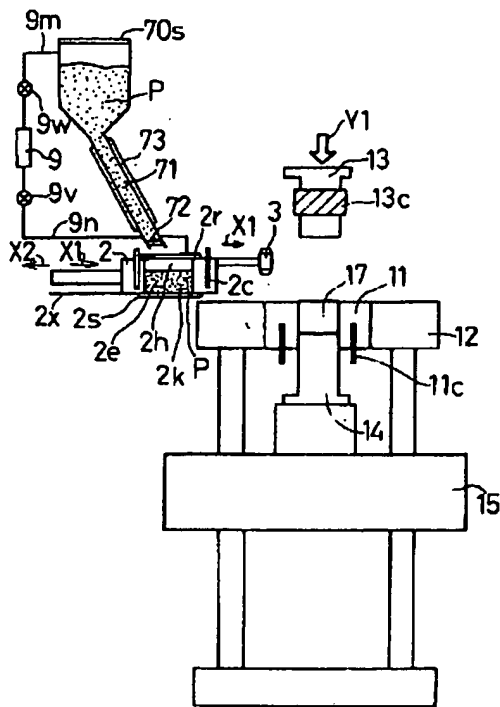


【図2】

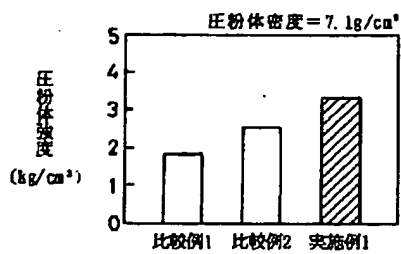




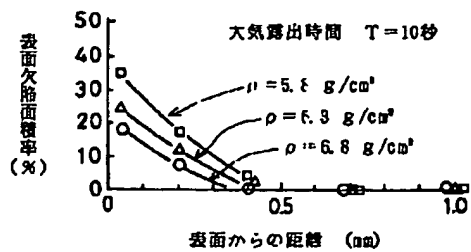
【図3】



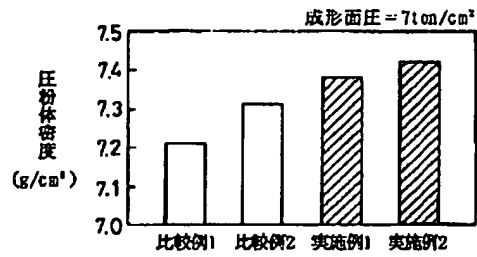
【図5】



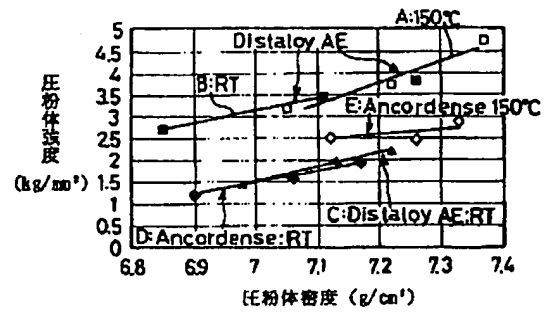
【図8】



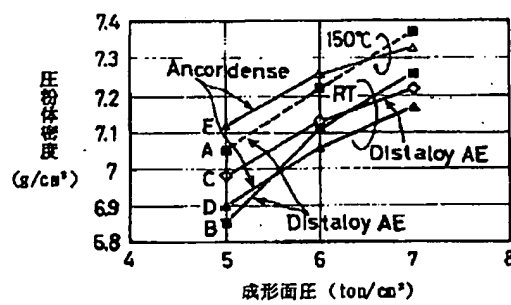
【図4】



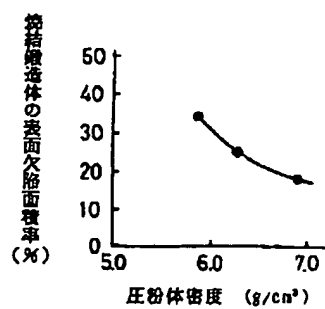
【図7】



【図6】



【図9】



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Japanese Kokai Patent Application  
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**POWDER MOLDING METHOD**

**Hiroshi Harada and Kazuhiko Takahashi**

UNITED STATES PATENT AND TRADEMARK OFFICE  
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POWDER MOLDING METHOD

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[There are no amendments to this patent.]

Claim

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A powder molding method characterized by the fact that the following steps of operation are performed in order:

a step in which a molding die for powder molding equipped with die surfaces that define the molding cavity and a molding powder substantially free of lubricant are used,

and in which a heating operation is performed to heat said molding die and said molding powder to within a temperature range of 150-400°C, and, at the same time, a coating operation is performed to coat said lubricant on the die surfaces of said molding die;

a step in which said molding powder heated to a temperature in said range is filled in the molding cavity of said molding die heated to a temperature in said range;

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[Numbers in right margin indicate pagination of the foreign text.]

and a step in which said molding powder filled in said molding cavity is compression-molded to form a green compact.

#### Detailed explanation of the invention

[0001]

##### Technical field of the invention

This invention pertains to a powder molding method for making compression-molding of powder to form green compact.

[0002]

##### Prior art

In the conventional powder metallurgy, zinc stearate or other lubricant is added in the molding powder so as to guarantee the lubrication property of the molding powder. US Patent No. 4,955,798 described a warm powder molding method characterized by the following facts: a molding powder for powder metallurgy and containing a solid lubricant is used; the molding powder is heated to a temperature in the range from room temperature to a temperature without melting of the solid lubricant, that is, in the range of 70-150°C, or preferably 70-120°C; and the heated molding powder is compression-molded using a molding die to form a green compact.

[0003]

Also, Japanese Kokai Patent Application No. Hei 5[1993]-271709 described a technology in which a lubricant for high-temperature use is added, and the lubricant is a product of a reaction among the following components: 10-30 wt% of C10-22 monocarboxylic acid, 10-30 wt% of C10-22 dicarboxylic acid, and 40-80 wt% of diamine represented by  $(CH_2)_x(CH_2)_2$  (x: 2-6). This type of lubricant can be used as a powder metallurgical lubricant at 370°C or lower.

[0004]

##### Problems to be solved by the invention

However, in order to increase the density of the powder particles themselves, one may increase the heating temperature of the powder. Yet when the molding powder is heated to 150°C or higher, fluidity of the lubricant added in the molding powder degrades significantly, and it is impossible to obtain a green compact with a high density. Even in the technology of said US Patent No. 4,955,798, although it is possible to guarantee fluidity of the molding powder if the temperature is lower than 150°C, when the temperature is over 150°C, the lubricant melts, and fluidity of the molding powder degrades significantly, so that it is impossible to fill the

powder in a powder box or the like, and this is unfavorable for continuous molding operation (unfavorable for productivity).

[0005]

For the technology described in Japanese Kokai Patent Application No. Hei 5[1993]-271709, as the melting point is higher than that of the conventional lubricant, it can be used at 370°C or lower, yet fluidity of the powder degrades at temperature of 150°C or higher. Due to the aforementioned facts, in the conventional warm powder molding method, the heating temperature of the powder is lower than 150°C.

[0006]

The objective of this invention is to solve the aforementioned problems of the prior art by providing a powder molding method preferred in improving the density and strength of the green compact.

[0007]

Means to solve the problems

In order to realize the aforementioned objective, the present inventors have performed extensive research. As a result of this research work, it was found that the molding powder can be compression-molded to form a green compact with improved density and strength when the following method is adopted: a molding powder substantially free of lubricant is used; both the molding powder and molding die are heated to a temperature in the range of 150-400°C, and a lubricant is coated on the die surfaces of the molding die; then the molding powder is compression-molded to form a green compact.

[0008]

That is, this invention pertains to a powder molding method characterized by the fact that the following steps of operation are performed in order: a step in which a molding die for powder molding equipped with die surfaces that define the molding cavity and a molding powder substantially free of lubricant are used, and in which a heating operation is performed to heat said molding die and said molding powder to a temperature in the range of 150-400°C, and, at the same time, a coating operation is performed to coat said lubricant on the die surfaces of said molding die; a step in which said molding powder heated to a temperature in said range is filled in the molding cavity of said molding die heated to a temperature in said range; and a step in which said molding powder filled in said molding cavity is compression-molded to form a green compact.

[0009]

#### Embodiment of the invention

A conventional die having die surfaces that define the molding cavity may be used as the molding die adopted in the method of this invention. A conventional metal powder substantially free of lubricant may be adopted as a molding powder adopted in the method of this invention. Consequently, the molding powder adopted in the method of this invention usually is not entirely free of lubricant. Conventional materials, such as iron based materials, Ni based materials, (O copper based materials, aluminum based materials, etc., may be used as the metal powder. As the metal powder, the conventional types, such as sprayed powder, electrolytic powder, reduced powder, etc., may be adopted.

[0010]

When the molding powder is heated to a high temperature, although improvement is realized with respect to the softening property and moldability of the molding powder, oxidation of the molding powder may also be accelerated. Also, if the heating temperature of the molding powder is low, although it is favorable in suppressing oxidation of the molding powder, softening property and moldability of the molding powder nevertheless degrade. Also, when the size of the powder particles of the molding powder is small, the specific surface area of the molding powder becomes larger, and the molding powder is prone to activation and oxidation.

[0011]

The heating temperature of the molding powder is determined in consideration of this fact. Depending on the particle size of the powder particles, the heating temperature of the molding powder has its upper limit set at 350°C, 300°C, 250°C, or 200°C, and has its lower limit set at 170°C, 200°C, or 230°C. However, this invention is not limited to said temperatures.

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[0012]

Also, if the heating temperature of the molding die becomes higher, although the moldability of the molding powder is improved, the lifetime of the molding die becomes shorter. This is undesired. On the other hand, if the heating temperature of the molding die becomes lower, although the lifetime of the molding die becomes longer, the moldability of the molding powder may degrade. The heating temperature of the molding die is determined in consideration of these facts. Depending on the type of the material of the molding die, the heating temperature of the molding die has its upper limit set at 350°C, 300°C, 250°C, or 200°C, and has its lower limit set at 170°C, 200°C, or 230°C.

[0013]

According to the method of this invention, when the molding powder is of iron type and the molding die is also of iron type, in consideration of the aforementioned facts, it is preferred that the molding powder and molding die be in the range of about 150-300°C. However, this invention is not limited to this range. Means for heating the molding powder and molding die include IR heating, heater, induction heating, joule heating, etc.

[0014]

According to the method of this invention, the molding die and molding powder are heated to a temperature in the range of 150-400°C. One may have the heating temperature of the molding die substantially equal to the heating temperature of the molding powder, or have them be different from each other. When the heating temperature of the molding powder is higher than the heating temperature of the molding die, the lifetime of the molding die can be guaranteed, while the moldability of the molding powder can be guaranteed. As a result, it is favorable in guaranteeing the density of the green compact.

[0015]

On the other hand, when the heating temperature of the molding die is higher than the heating temperature of the molding powder, transfer of heat from the molding powder to the molding die is suppressed. Consequently, this is favorable in maintaining the temperature of the molding powder. As a result, it is favorable in guaranteeing the density of the green compact. In particular, as heat of the outer layer portion of the green compact is guaranteed, it is favorable for maintaining the surface temperature of the green compact, and thus for forming a finer surface of the green compact.

[0016]

The atmosphere for heating the molding powder can be selected corresponding to the type of the molding powder. It may be performed in the ambient atmosphere or in a non-oxidative atmosphere. Examples of the non-oxidative atmosphere include reduced pressure atmosphere, vacuum atmosphere, nitrogen atmosphere, rare gas atmosphere, etc. Also, a reductive atmosphere may be adopted. The same is true for the atmosphere for heating the molding die. According to the method of this invention, a lubricant is coated on the die surfaces of the molding die. As a result, friction between the molding powder and the die surfaces of the molding die can be reduced, and filling of the molding powder becomes easier. Conventional types of lubricants can be adopted as the lubricant in this case. One may adopt a solid lubricant

or adopt a lubricant prepared by dispersing or dissolving a solid lubricant in a solvent. Examples of the lubricants that may be adopted include zinc stearate based lubricants, graphite based lubricants, boron nitride based lubricants, metal soap based lubricants, etc. Examples of solvents include water, alcohol, etc. As far as the coating means is concerned, one may adopt spray coating, brushing, dipping, and other conventional methods.

[0017]

According to the method of this invention, there is a step of heating operation in which the molding die and molding powder are heated to a temperature range of 150-400°C, and there is a step of coating operation in which a lubricant is coated on the die surfaces of the molding die. In this case, one may perform the coating operation after the heating operation, or perform the heating operation after the coating operation, or perform the heating operation and coating operation at the same time.

[0018]

#### Application examples

In the following, this invention will be explained in more detail with reference to application examples. First of all, explanation will be made on molding die (1) with reference to Figure 1. Molding die (1) has die mold (11) made of a metal in ring shape and functioning as a fixed die with a die hole, die holder (12) made of a metal in ring shape and holding the peripheral portion of die mold (11), upper punch die (13) made of a metal and capable of lifting with respect to die mold (11), and lower punch die (14) made of a metal and fit to the die hole of die mold (11). Said lower punch die (14) is held on base portion (15).

[0019]

Molding cavity (17) is defined with the die surface of the die hole of die mold (11), the die surface of lower punch die (14), and the die surface of upper punch die (13). Upper punch die (13) is equipped with first heater (13c) for heating it. Die mold (11) is equipped with second heater (11c) for heating it. In addition, movable powder box (2) that can move along guide (2x) is equipped above die holder (12) and die mold (11). Powder box (2) has containing chamber (2e) that contains the molding powder. Containing chamber (2e) has upper surface opening (2h) and lower surface opening (2k). On powder box (2), spray nozzle (3) for spraying the lubricant is equipped via arm (3m). Powder box (2) has third heater (2c) for heating molding powder P in containing chamber (2e).



[0020]

Also, powder feeder (7) is set near molding die (1). Powder feeder (7) is composed of powder container (70) that contains molding powder P, feeding channel (71) that is set extending from powder container (70) downward to above containing chamber (2e) of powder box (2) in standby state, valve (72) for opening/closing feeding channel (71), and fourth heater (73) for heating molding powder P in feeding channel (71). Said first heater (13c), second heater (11c), third heater (2c), and fourth heater (73) are controlled with a temperature controller not shown in the figure, and they are maintained at their respective target heating temperatures.

[0021]

When this molding die is used in this application example, die mold (11) is heated to a prescribed temperature range by means of second heater (11c), and upper punch die (13) is heated to a prescribed temperature range with first heater (13c). Also, molding powder P of powder container (70) is heated to a prescribed temperature range by means of fourth heater (73) of powder feeder (7). In this application example, the target heating temperature for molding die (1) is in the range of 150-400°C, and the target heating temperature for molding powder P is in the range of 150-400°C.

[0022]

As shown in Figure 1, powder box (2) is set in standby state at standby position M separated from molding cavity (17). When valve (72) of powder feeder (7) is turned ON, heated molding powder P falls naturally from powder feeder (7), and it is fed to containing chamber (2e) of powder box (2). The molding powder in containing chamber (2e) of powder box (2) is heated with third heater (2c), and it is set in standby state within a prescribed temperature range in containing chamber (2e).

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[0023]

At a prescribed time, as shown in Figure 2(A), powder box (2) is driven to move in the direction indicated by arrow X1 by means of cylinder (2p). Consequently, as shown in Figure 2(B), spray nozzle (3) becomes opposite to molding cavity (17) and upper punch die (13), and powder box (2) is stopped at the position. In this state, spray nozzle (3) is turned ON, lubricant L is sprayed from spray nozzle (3) in the upward and downward directions. As a result, lubricant L is coated on the die surface of die mold (11), the die surface of lower punch die (14), and the die surface of upper punch die (13). That is, "die lubricating" operation is carried out as lubricant L is coated on the die surfaces of molding die (1).

[0024]

In this application example, because die mold (11) and upper punch die (13) are heated to a high temperature, liquid lubricant L is quickly dried under their heat. Consequently, a lubricant film is laminated on the die surface of die mold (11), the die surface of lower punch die (14), and the die surface of upper punch die (13). Consequently, it is possible to omit or simplify the drying operation for drying liquid lubricant L, and this contributes to increase of the production efficiency.

[0025]

After end of coating as the "die lubricating operation", powder box (2) is further driven to move in the direction indicated by arrow X1. As shown in Figure 2(C), lower opening (2k) of containing chamber (2e) of powder box (2) becomes opposite to molding cavity (17). Then, molding powder P in containing chamber (2e) of powder box (2) falls naturally under its own weight to fill molding cavity (17). After end of filling, powder box (2) is driven to move in the direction indicated by arrow X2 and is returned to standby position M. Then, upper punch die (13) is lowered in the direction indicated by arrow Y1, and it is pressed into molding cavity (17) to form a green compact. Therefore molding powder P in molding cavity (17) is compression-molded and a green compact is molded.

[0026]

In this application example, friction between molding powder P in molding cavity (17) and the die surface of molding die (1) is reduced by said lubricant film. As a result, filling property and compression-molding property of molding powder P are improved. After molding of said green compact, the following steps of operation are performed: a sintering step in which the green compact is heated to the sintering temperature range to form a sintered body, and a forging step in which the sintered body is hot forged with a forging die to form a sintered forged body.

[0027]

In this application example, when molding powder P and molding die (1) are heated, the heating temperature of molding powder P and the heating temperature of molding die (1) may be substantially identical to each other. Also, one may have the heating temperature of molding powder P higher than the heating temperature of molding die (1). In addition, one may have the heating temperature of molding die (1) higher than the heating temperature of molding powder P. When the heating temperature of molding powder P is higher than the heating temperature of

molding die (1), it is favorable for guaranteeing the moldability of molding powder P, and it is thus favorable for guaranteeing the density of the green compact.

[0028]

When the heating temperature of molding die (1) is higher than the heating temperature of molding powder P, even when molding powder P is in contact with the die surface of molding die (1), it is possible to suppress escape of heat of molding powder P to molding die (1), so that it is favorable for maintaining the temperature of molding powder P. In this sense, it is favorable for guaranteeing the density of the green compact. Especially, it is possible to guarantee the temperature of the surface layer portion of the green compact in contact with the die surface of molding die (1). Consequently, this is favorable for maintaining the surface temperature of the green compact. This is favorable for avoiding or reducing the surface pores.

[0029]

In the aforementioned example, coating operation by spraying lubricant L and loading operation by having molding powder P in powder box (2) naturally fall into molding cavity (17) are carried out while powder box (2) is stopped. However, this invention is not limited to this scheme. It is also possible to perform the aforementioned coating operation and loading operation while powder box (2) is driven to move at a low velocity instead of stopping it. In the aforementioned application example, there is a forging step of operation in which the sintered body prepared by sintering the green compact is forged. However, this invention is not limited to this scheme. One may also omit the forging step, depending on the specific application of the green compact.

[0030]

Also, one may perform the operation in the example shown in Figure 3. In this case, in addition to shutoff lid (2r) for opening/closing upper opening (2h) of containing chamber (2e) of powder box (2), and shutoff lid (2s) for opening/closing lower opening (2k), containing chamber (2e) is connected to evacuating device (9) through evacuating channel (9n) and shutoff valve (9v). As evacuating channel (9n), a rubber hose, a bellows tube, or other stretchable, bendable and flexible tube may be adopted. In this way it allows movement of powder box (2). For example, a vacuum pump may be used as evacuating device (9).

[0031]

Then, as the molding powder in containing chamber (2e) is heated with third heater (2c), containing chamber (2e) is in reduced pressure state or vacuum state by means of evacuating

device (9) and it stands by in this state. At end of the standby time, powder box (2) is driven to move in the direction indicated by arrow X1. When powder box (2) reaches a prescribed position, shutoff lid (2r) and shutoff lid (2s) are opened, or, in some cases, only shutoff lid (2s) is opened, so that molding powder P in powder box (2) falls into molding cavity (17). As a result, because containing chamber (2e) is under a reduced pressure or in vacuum state in standby, oxidation of molding powder P at a high temperature can be suppressed. This is favorable.

[0032]

Because it is favorable for suppressing oxidation of molding powder P, it is possible to further increase the heating temperature of molding powder P. As a result, the density of the green compact is further increased, and it contributes to guarantee the quality of the green compact, sintered body and sintered forged body. In addition, as shown in Figure 3, one may also adopt the following scheme: shutoff (70s) for opening/closing powder container (70) of powder feeder (7) is set on said powder container, and, at the same time, powder container (70) is connected to evacuating device (9) through evacuating channel (9m) and shutoff valve (9w). In this case, it is possible to suppress oxidation of the molding powder at a high temperature in powder container (70).

[0033]

(Test examples)

In order to check the effects of the method of this invention, the following tests were performed.

(Test Example 1)

① In this test, a mixture of iron based powder and graphite powder was used as the molding powder, and the molding surface pressure for molding the green compact was set at the same value of 7 ton/cm<sup>2</sup> in both the application examples and comparative examples. In each application example and comparative example, a green compact was formed, and the density of the green compact was measured. Measurement of density is based on the standard of Japanese Powder Metallurgical Industry Association (JPMA M 01-1992). Also, in both application examples and comparative examples, the proportion of the graphite powder in the molding powder is 5 wt%.

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[0034]

The iron based powder adopted in both the application examples and comparative examples has a commercial name of Distaloy AE (Hoganas Co). Target composition of Distaloy

AE by weight is of Fe, 4% Ni, 1.5% Cu and 0.5% Mo. Particle size distribution of Distaloy is as follows: 8% of particles with size of 150  $\mu\text{m}$  or larger, 20% of particles with size of 45  $\mu\text{m}$  or smaller, and balance of particles with size of 45 to 150  $\mu\text{m}$ .

[0035]

In Application Example 1, said molding powder without lubricant is used. Together with die lubrication, in a state in which the molding temperature is set at 150°C, that is, in a state in which target temperature of both the molding powder and the molding die is set at 150°C, a green compact is molded. In Application Example 2, said molding powder without lubricant is used. In the same way as aforementioned, together with die lubrication, in a state in which the molding temperature is set at 180°C, that is, in a state in which the temperature of both the molding powder and the molding die is set at 180°C, a green compact is molded.

[0036]

In Comparative Example 1, a green compact is molded from a molding powder, which has zinc stearate added as lubricant to said iron based powder, in a state in which the molding temperature is kept at room temperature, that is, in a state in which the temperature of both the molding powder and the molding die is kept at room temperature. In Comparative Example 1, with the amount of the molding powder taken as 100%, 0.5% of zinc stearate is added. In Comparative Example 2, a green compact is molded from a molding powder, which has lubricant (lubricant described in US Patent No. 4,955,798) used for warm molding added to said iron based powder, in a state in which the molding temperature is set at 150°C, that is, in a state in which the temperature of both the molding powder and the molding die is set at 150°C. In Comparative Example 1, with the amount of the molding powder taken as 100%, 0.5% of lubricant used for warm molding is added.

[0037]

Table 1

	Lubrication	Molding temperature
Comparative Example 1	Zinc stearate	Room temperature
Comparative Example 2	Lubricant for warm molding	160°C
Application Example 1	Die lubrication	150°C
Application Example 2	Room temperature	180°C

Test results of density measurement of the green compacts are illustrated in Figure 4. As can be seen from Figure 4, even with the same molding surface pressure, density of the green compacts in Comparative Examples 1 and 2 is lower, while density of the green compacts formed in Application Examples 1 and 2 is higher. Especially in Application Example 2, with molding temperature set at 180°C, density of the green compact is the highest. It can be seen that even if the molding surface pressure is kept the same ( $= 7 \text{ ton/cm}^2$ ) as the green compact is formed, by using the method in this application example, density of the green compact can be increased. The reason is believed to be a good fineness of the pores as no lubricant is used.

[0038]

② Furthermore, when density of green compacts is set at  $7.1 \text{ g/cm}^3$ , flexural strength of the green compacts in the application examples and comparative examples was measured. Measurement of flexural strength is based on the standard of Japanese Powder Metallurgical Industry Association (JPMA M 09-1992). Test results are illustrated in Figure 5. As can be seen from Figure 5, strength of the green compacts in Comparative Examples 1 and 2 is lower, while the green compact formed in Application Example 1 has a high strength over  $3 \text{ kg/cm}^2$ . It can be seen that even if density of the green compacts is kept the same, by using the method in this application example, strength of the green compact can be increased. The reason is believed to be that solidification (entanglement of powder) among the powder particles becomes stronger as no lubricant is used.

[0039]

(Test Example 2)

① In this test, as green compacts were formed by compression-molding the molding powder, which is formed from a mixture of iron based powder and graphite powder, by changing the molding surface pressure from  $5 \text{ ton/cm}^2$  to  $6 \text{ ton/cm}^2$  to  $7 \text{ ton/cm}^2$ , changes in density of the green compacts were studied in both the application examples and comparative examples. Measurement of density is the same as aforementioned, and it is based on the standard of Japanese Powder Metallurgical Industry Association (JAMA M 09-1992). Also, in both said application examples and comparative examples, the proportion of the graphite powder in the molding powder is 5 wt%.

[0040]

In Application Example A, said Distaloy AE (Hoganas Co) was adopted as the iron based powder, and target temperature of both the powder and the molding die was set at 150°C. Target composition of Distaloy AE is the same as aforementioned, that is, the composition by weight is

of Fe, 4% Ni, 1.5% Cu and 0.5% Mo. Test results in Application Example A are shown in Figure 6. The abscissa in Figure 6 represents the molding surface pressure, and the ordinate represents density of the green compacts. RT stands for room temperature in Figure 6.

[0041]

In Comparative Example B, Distaloy AE (Hoganas Co) was used as the iron based powder, and temperature of both the powder and the molding die was set at room temperature. In Comparative Example C, Distaloy AE (Hoganas Co) with zinc stearate added in was used as the iron based powder, and temperature of both the molding powder and the molding die was set at room temperature. In Comparative Example D, Ancordense (Hoganas Co), which is prepared by adding a lubricant for high-temperature use that does not melt even in the high-temperature range instead of zinc stearate, was used as the iron based powder. In this comparative example, temperature of both the powder and the molding die was set at room temperature.

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[0042]

In Comparative Example E, Ancordense was used as the iron based powder, and the temperature of both the powder and the molding die was set at room temperature. As can be seen from Figure 6, in Comparative Examples C, D and B, in which the green compacts are molded from the powder and the molding die at temperature in the room-temperature range, density of the green compacts is low. Also, in Comparative Example E, in which a lubricant for high-temperature use is added to the molding powder, density of the green compact is high as molding surface pressure is in the range of 5-6 ton/cm<sup>2</sup>, while as molding surface pressure approaches 7 ton/cm<sup>2</sup>, the effect of increase in density levels off.

[0043]

On the other hand, in Application Example A, in which target temperature of both the powder and the molding die is set at 150°C, even when the molding surface pressure is set at 7 ton/cm<sup>2</sup>, the effect of increase in density does not level off. That is, as seen in Figure 6, density in Application Example A is higher than that in Comparative Example E if molding surface pressure is at 7 ton/cm<sup>2</sup>. The reason is believed to be as follows: in the case that molding surface pressure is high and lubricant is contained, the portion corresponding to the lubricant is not substantially filled with metal powder; instead, it substantially becomes pores, so that a high-density green compact is not formed. On the other hand, when there is no lubricant, there is no such problem.

② Also, for the green compacts obtained in said Application Examples A-E, testing was carried out to study their flexural strength. Measurement of the flexural strength is carried out on

the base of the standard of Japanese Powder Metallurgical Industry Association (JPMA M 09-1992). The results of the test are shown in Figure 7. In Figure 7, the abscissa represents the density of the green compact, and the ordinate represents the strength of the green compact. In Application Example A, when the density of the green compact is as high as in the range of 7.1-7.3 g/cm<sup>3</sup>, the strength of the green compact is increased to about 3-4.7 kgf/mm<sup>2</sup>. That is, in Application Example A, the slope of increase of the characteristics curve is large.

[0044]

On the other hand, in Comparative Example E, even when the density of the green compact is increased to 7.1-7.3 g/cm<sup>3</sup>, the strength of the green compact is still merely a little over 2.5 kgf/mm<sup>2</sup> with little increase. That is, in Comparative Example E, the slope of rise of the characteristic curve is small.

③ In use, said green compact is sintered to form a sintered body. In order to further increase the density, it is preferred that forging be performed after sintering to form a sintered forged body under high pressure. In the practical operation in this case, it is necessary to transport the sintered body from the sintering step to the forging step. Consequently, the sintered body makes contact with the ambient atmosphere during the process of said transportation. If the sintered body has a high degree of exposure to the ambient atmosphere, when it is forged, surface defects are developed on the surface of the sintered forged body, and this is undesired with respect to maintaining the strength of the sintered forged body.

[0045]

In the test, after a sintered body is formed by sintering said green compact in the sintering temperature range (1120-1150°C), the sintered body is exposed to the ambient atmosphere for a prescribed time T (T=10 sec). Then, the sintered body is hot forged to form a sintered forged body. Then, the proportion of area of the surface defects of the sintered forged body is measured. The results are shown in Figure 8, with density  $\rho$  of the green compact taken as a parameter.

[0046]

In Figure 8, the abscissa represents the distance from the surface of the sintered forged body, and the ordinate represents the proportion of area of the surface defects. It can be seen from Figure 8 that the proportion of area of the surface defects depends significantly on the density of the green compact. That is, when the green compact has a low density ( $\rho=5.8$ ), the proportion of area of the surface defects of the sintered forged body is high. It is believed that in this case, fineness of the surface decreases, and the sintered body is prone to surface oxidation. On the other hand, if the green compact has a high density ( $\rho=6.8$ ), the proportion of area of the



surface defects becomes smaller. It is believed that in this case, fineness of the surface becomes better, and it becomes harder for surface oxidation of the sintered body to make progress.

[0047]

Consequently, the method of this invention can effectively increase the density of the green compact, and it is preferred in reducing the surface defects of the sintered forged body. As a result, it is favorable for increase in the strength of the sintered forged body. Figure 9 is a diagram illustrating the relationship between the density of the green compact and the proportion of area of the surface defects on the sintered forged body. It can be seen from the characteristic curve in Figure 9 that when the density of the green compact becomes lower, the proportion of area of the surface defects of the sintered forged body becomes larger. On the other hand, when the density of the green compact becomes higher, the proportion of area of the surface defects of the sintered forged body becomes smaller.

[0048]

(P.S.) From the aforementioned application examples, the following technical ideas have been obtained.

① According to Claim 1, the powder molding method is characterized by the fact that the molding powder and the molding die are substantially at the same temperature.

② According to Claim 1, the powder molding method is characterized by the fact that the temperature of the molding powder is lower than the temperature of the molding die.

③ According to Claim 1, the powder molding method is characterized by the fact that the temperature of the molding powder is higher than the temperature of the molding die.

④ A Powder molding method characterized by the fact that a powder box that can be driven to move between a standby position away from the molding die to a position facing the molding die is used, and the interior of the standby powder box can be maintained in reduced pressure state or vacuum state.

⑤ A method for manufacturing a sintered forged body characterized by the fact that the following steps of operation are performed in order: a step in which a molding die for powder molding equipped with die surfaces that define the molding cavity and a molding powder substantially free of lubricant are used and in which a heating operation is performed to heat said molding die and said molding powder to within a temperature range of 150-400°C, and, at the same time, a coating operation is performed to coat said lubricant on the die surfaces of said molding die; a step in which said molding powder is filled in the molding cavity of said molding die; a step in which said molding powder filled in said molding cavity is compression-molded to form a green compact; a step in which the green compact is sintered to form a sintered body; and

a step in which the sintered body is forged to form the desired sintered forged body. This method can realize a high density of the green compact. Consequently, it can reduce surface defects so as to guarantee the strength of the sintered forged body.

[0049]

#### Effect of the invention

According to the method of this invention, the following steps of operation are performed in order: a step in which a molding die for powder molding equipped with die surfaces that define the molding cavity and a molding powder substantially free of lubricant are used, and in which a heating operation is performed to heat said molding die and said molding powder to within a temperature range of 150–400°C, and, at the same time, a coating operation is performed to coat said lubricant on the die surfaces of said molding die; and a step in which said molding powder is filled in the molding cavity of said molding die. As a result, it is favorable for increasing the density and strength of the green compact.

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[0050]

Increase in the density of the green compact is favorable in reducing the pores on the surface of the green compact. Consequently, when the green compact is sintered and forged, surface defects on the surface of the sintered forged body can be reduced effectively, and this is favorable for further increase in the strength of the sintered forged body.

#### Brief description of the figures

Figure 1 is a diagram illustrating the constitution of an application example.

Figure 2 is a diagram illustrating the constitution of the various steps of operation in the application example.

Figure 3 is a diagram illustrating the constitution of other examples.

Figure 4 is a graph illustrating the density of the green compact when the molding surface pressure is at 7 ton/cm<sup>2</sup>.

Figure 5 is a graph illustrating the strength of the green compact when the density of the molding is 7.1 g/cm<sup>3</sup>.

Figure 6 is a graph illustrating the relationship between the molding surface pressure and the density of the green compact.

Figure 7 is a graph illustrating the relationship between the density of the green compact and the strength of the green compact.

Figure 8 is a graph illustrating the relationship between the distance from the surface of the sintered forged body and the proportion of area of surface defects of the sintered forged body.

Figure 9 is a graph illustrating the relationship between the density of the green compact and the proportion of area of the surface defects of the sintered forged body.

#### Explanation of symbols

- 1 Molding die
- 11 Die mold
- 13 Upper punch die
- 17 Molding cavity
- 11c, 12c, 13c, 73 Heater
- 2 Powder box
- 3 Spray nozzle

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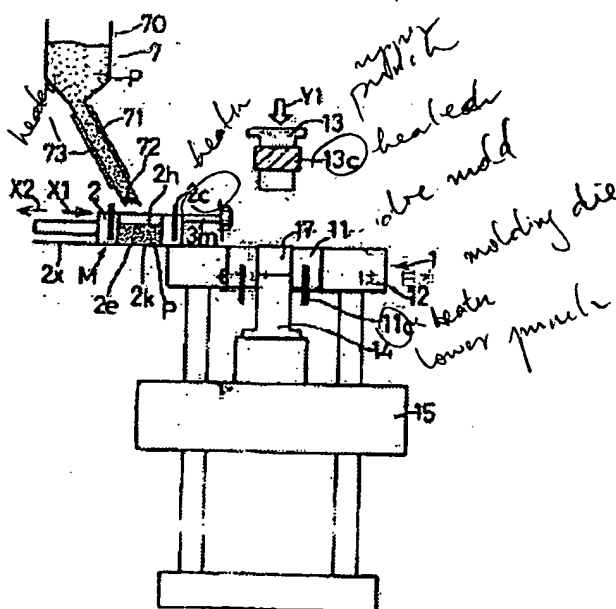


Figure 1

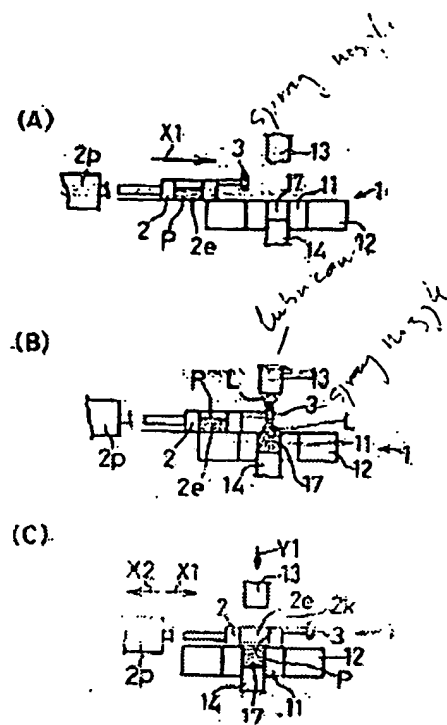


Figure 2

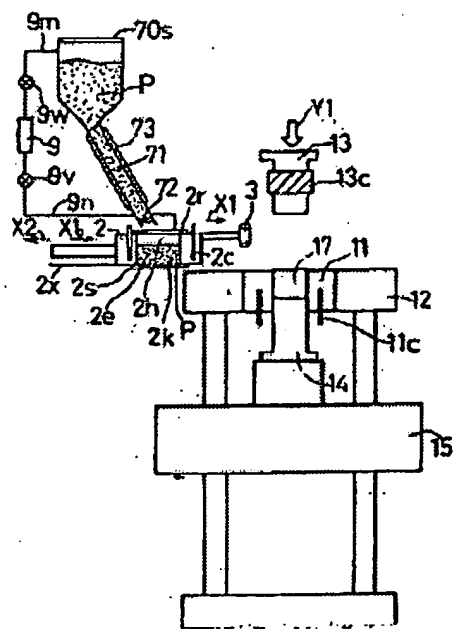


Figure 3

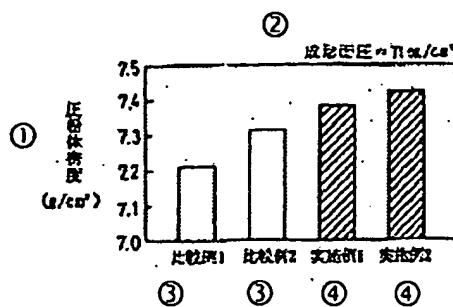


Figure 4

- Key: 1 Density of green compact  
 2 Molding surface pressure  
 3 Comparative Example  
 4 Application Example

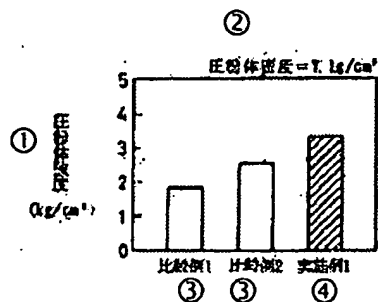


Figure 5

- Key: 1 Strength of green compact  
 2 Density of green compact  
 3 Comparative Example  
 4 Application Example

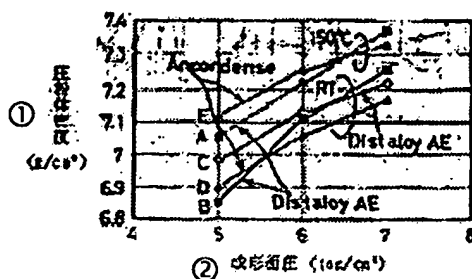


Figure 6

- Key: 1 Density of green compact  
 2 Molding surface pressure

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